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THE CONSTITUTION OF LEAD OXIDE-SILICA GLASSES: I, ATOMIC ARRANGEMENT*

By George J. Bair

Abstract

A series of lead oxide-silica glasses varying in composition from 10 to 60 mol % of PbO was prepared and diffraction patterns were obtained. The observed intensity curves derived from these patterns were matched with curves calculated from assumed structures by means of the scattering equation of Zernicke and Prins. The atomic arrangement finally derived consists of a continuous, randomly oriented, three-dimensional network of silicon-oxygen tetrahedrons and lead atoms. There was no indication of molecular or aggregate formation and no discrete atomic grouping of this nature was postulated to explain the X-ray evidence. The study of this series of glasses further substantiates the results obtained by Warren and Loring for soda-silica glasses and is in accord with Zachariasen's picture of glass.

I. Introduction

The constitution of glass has been a subject of much study and speculation, and the discussion of the true nature of glass has become especially fervent during the last ten years. From thermodynamic considerations and the comparison of the cooling curves of glassy and crystallizing materials, glass has been termed a supercooled liquid.[1] It passes through the liquidus temperature with a continuous change in its properties. Careful measurement of the properties of glasses over the full range of temperature, however, shows unique breaks in their property-temperature curves at about 100°C below the liquidus.[2] Many glass technologists consider that these breaks are due to a rapidly increasing inability of the glass to maintain an equilibrium degree of association as it is cooled through this critical temperature range. They explain that the break in the property-temperature curve of a glass results from the intersection of a curve representing the change in properties at the high temperatures where equilibrium is readily established, with the curve representing low temperature conditions where equilibrium is not obtained.[3]

Other investigators attribute true significance to the "transformation point" and postulate that this is the temperature at which, on cooling, the glass changes from a viscous liquid to a solid.[4]

From purely theoretical considerations of interatomic forces, glasses have been described as a continuous, distorted network of atoms.[5] Some consider the presence of aggregates which are randomly oriented with respect to each other. These aggregates may possess a definite orderly structure but are exceedingly small.[6]

The X-ray has supplied the only direct evidence of atomic arrangement in glass. At first the diffraction pattern obtained from glass was interpreted to indicate crystallite formation.[7] A

* These papers (Parts I and II) were prepared from material presented in a thesis submitted in partial fulfillment of the requirements for a degree of Doctor of Science, in the field of Ceramics, Department of Mining and Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass., while on leave of absence from the Pennsylvania State College, State College, Pa.

Presented at the Annual Meeting, American Ceramic Society, Columbus, Ohio, March 31, 1936 (Glass Division). Received July 31, 1936.

[1] (*a*) G. Tammann, The Glassy State. Leopold Voss, Leipzig, 123 pp. (1933).

(*b*) G. W. Morey, "Constitution of Glass," *Jour. Amer. Ceram. Soc.*, **17** [11] 315–28 (1934).

[2] (*a*) A. L. Day and E. T. Allen, "Isomorphism and Thermal Properties of Feldspars," *Amer. Jour. Sci.*, **19**, 93 (1905).

(*b*) A. Q. Tool and C. G. Eichlin, "Variations Caused in Heating Curves of Glass by Heat Treatment," *Jour. Amer. Ceram. Soc.*, **14** [4] 276–308 (1931); *Bur. Stand. Jour. Research*, **6** [4] 523–52 (1931).

(*c*) C. G. Peters and C. H. Cragoe, "Measurements of the Thermal Dilatation of Glass," *Bur. Stand. Sci. Paper*, No. 393 (1920); abstracted in *Jour. Amer. Ceram. Soc.*, **3** [10] 852 (1920).

(*d*) M. Fulda, "Electrical Conductivity of Glasses," *Sprechsaal*, **60** [42] 769–72 (1927); *Ceram. Abs.*, **7** [3] 150 925 (1928).

[3] (*a*) J. T. Littleton, "Critical Temperatures in Silicate Glasses," *Ind. Eng. Chem.*, **25** [7] 748–55 (1933); *Ceram. Abs.*, **12** [10–11] 360 (1933).

(*b*) O. Koerner, H. Salmang, and W. Lerch, "Dilatometric Measurements on the Glassy State," *Sprechsaal*, **65**, 925 (1932).

[4] E. Berger, "Contributions to Theory of Glass Formation and the Glassy State," *Jour. Amer. Ceram. Soc.*, **15** [12] 647–77 (1932).

[5] W. H. Zachariasen, "Atomic Arrangement in Glass," *Jour. Amer. Chem. Soc.*, **54** [10] 3841–51 (1932); *Ceram. Abs.*, **12** [4] 145 (1933).

[6] (*a*) W. Rosenhain, "Structure and Constitution of Glass," Chapt. XII, pp. 166-86 in The Constitution of Glass, 192 pp. Published by Society of Glass Technology, Sheffield, England (1927).

(*b*) G. Hagg, "Vitreous State," *Jour. Chem. Phys.* [London], **3** [1] 42–49 (1935); *Ceram. Abs.*, **14** [7] 161 (1935).

[7] (*a*) C. W. Parmelee, G. L. Clark, and A. E. Badger, "Diffractions of X-Rays by Ordinary Glass Subjected to Various Treatments," *Jour. Soc. Glass Tech.*, **13** [52] 285–90 (1929); *Ceram. Abs.*, **9** [5] 339 (1930).

(*b*) J. T. Randall, H. P. Rooksby, and B. S. Cooper,

more recent analysis, however, does not require the presence of aggregates or crystallites to explain the position and breadth of the band on the diffraction pattern but indicates that a continuous, randomly oriented network of atoms may fully describe the structure.[8]

In view of this recent X-ray interpretation from which a more definite picture of the structure of glass was obtained than had heretofore been possible, it seemed desirable to attempt a correlation of this atomic arrangement with the physical properties of the glass. It was proposed then to make a series of simple glasses and investigate their structures with X-rays. The physical properties of these glasses in the annealing range were then to be studied and correlated if possible with the structures determined.

A series of lead oxide-silica glasses was selected for this study because workable glasses are formed over a wide range of composition and because of the high X-ray scattering power of lead atoms which would permit more definite location in the structure.

The first part of this investigation (presented here as Part I) is the study of the atomic arrangement of these lead oxide-silica glasses by means of the X-ray.

II. Preparation of Glasses

Seven glasses were prepared, ranging in composition from 10 to 70 mol % of lead oxide. "Baker's analyzed" red lead containing less than 0.06% total impurities and purified quartz containing less than 0.25% total impurities† were used in preparing the glasses. A melting procedure suggested by Dr. Morey was followed as closely as possible.

One important digression from Morey's technique was the use of clay crucibles instead of platinum. Batches of 300 grams required large crucibles, and because there was a possibility of ruining the platinum by contaminating it with lead, the cost of platinum crucibles was prohibitive. Clay crucibles were made of pure kaolin and pure kaolin grog and were fired to 1600°C. These were dense and resistant to attack. For the high silica melts, the kaolin body was reinforced with 40% of fused alumina.

Before proportioning the batches, the silica was calcined at 1500°C for five hours to convert it to cristobalite. A base glass consisting of equimolecular proportions of PbO and SiO_2 was proportioned, melted, and quenched in water. The proper amount of lead oxide or silica was then added to this powdered base glass to give the series of glasses containing 30, 40, 50, 60, and 70 mol % of PbO. The batches were fritted, dried, ground, and remelted three times. Care was taken in the third and final melting to fine the glass completely. The temperature was then dropped to a good working temperature for drawing the specimens.

For the X-ray study, small slabs of glass were required. Because of the high absorptive power of the Pb atom, the slabs had to be very thin. Preliminary calculation of the linear adsorption coefficients to obtain values of optimum thickness for the best diffraction patterns indicated that the slab thicknesses should range between 0.004 to 0.0008 centimeter. The simplest way to obtain glasses of this thickness was to gather the molten glass on the end of a small fused silica tube and blow it into bubbles. These were broken and pieces of proper thickness were selected. After satisfactory specimens were obtained, the remaining glass was worked out of the crucible and drawn into fibers for the other tests.

For glasses containing less than 30% of lead oxide, a different melting procedure was required. The fining temperatures for these glasses were above 1600°C. A base glass containing 30 mol % of lead was prepared as described above. Proper silica addition was made and small 50-gram batches were melted in an induction furnace. In this case, single melts were made and the glass was cooled in the crucible. The outside portion of the glass and crucible were chipped away and a thin section of the center was prepared on a lap for the X-ray samples. The remainder of the piece was pulverized for analysis.

With the exception of glasses Nos. 10 and 20, all the glasses were readily worked and shaped. As the lead-oxide content increased, the glasses were more readily melted and their viscosity range became quite short. Glass No. 70 devitrified

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"Structure of Glasses; the Evidence of X-Ray Diffraction," *Jour. Soc. Glass Tech.*, **14** [54] 219–29 (1930); *Ceram. Abs.*, **9** [11] 926 (1930).

[8] (*a*) B. E. Warren, "X-Ray Diffraction of Vitreous Silica," *Z. Krist.*, **86**, 349 (1933).

(*b*) B. E. Warren, "X-Ray Determination of Structure of Glass," *Jour. Amer. Ceram. Soc.*, **17** [8] 249–54 (1934).

(*c*) B. E. Warren and A. D. Loring, "X-Ray Diffraction Study of Structure of Soda-Silica Glass," *ibid.*, **18** [9] 269–76 (1935).

† Obtained through the courtesy of Corning Glass Works.

easily, and to avoid any possibility of incipient devitrification during test, it was decided to omit this glass from the series.

The glasses were carefully analyzed for silica, iron, and aluminum, and the lead was determined by difference. The results are given in Table I.

TABLE I

CHEMICAL ANALYSES OF LEAD OXIDE-SILICA GLASSES

Glass No.	Weight percentage			Mol percentage		
	PbO	SiO_2	Al_2O_3–Fe_2O_3	PbO	SiO_2	Al_2O_3–Fe_2O_3
10	28.0	71.6	0.4	9.5	90.2	0.3
20	47.0	52.0	1.0	19.9	79.2	.9
30	62.6	37.0	0.4	31.2	68.4	.4
40	71.1	28.6	0.3	39.9	59.8	.3
50	79.4	20.3	0.3	49.8	49.8	.4
60	84.6	15.0	0.4	60.0	39.4	.6

III. Experimental Procedure

The X-ray study of the atomic arrangement in the glasses in the present investigation was conducted in the same manner as the previous glass studies by Warren and his associates.[8]

Diffraction patterns of the glasses were obtained by means of a Müller tube, with a copper target, and a vacuum camera. The beam of the X-rays was monochromatized by reflecting the Kα-line from a sodium-chloride crystal. After passing through a window of aluminum foil and a collimating tube, the purified beam struck the specimen. The slab of glass was cemented to a cap screwed onto the end of the collimating tube. This placed it in the exact center of the camera, the radius of which was 4.45 centimeters. The X-ray film was clamped to the cylindrical wall of the camera with brass rings. The primary beam passed through a hole in the film and out of the camera through an aluminum window. The camera was fitted with a vacuum-tight top and during operation a vacuum was maintained by means of a pump.

For this investigation, the tube was operated at 15 milliamperes and a 30-kilovolt peak. Thirty-hour exposures were sufficient to give distinct patterns. The patterns obtained are shown in Fig. 1. They are arranged in order of increasing PbO content with the pattern for pure vitreous silica at the top. There are several significant facts readily recognized in these patterns. With increasing lead-oxide content, there is a continuous sharpening of the principal band; the position of the band gradually changes to larger angles and a second, very diffused band becomes quite distinct.

A quantitative measure of the degree of blackening and, hence, the amount of scattering for each value of θ was obtained by means of a Kipp and Zonen microphotometer. The microphotometer curves were corrected for general scattering, and the comparative intensity curves were calculated by determining the logarithm of the ratio of total blackening, D_0, to the difference, D_x, between this total blackening and the observed blackening for the various values of $\frac{\sin\theta}{\lambda}$. The term, $\log \frac{D_0}{D_x}$, is proportional to the intensity of scattering at the particular angle considered. The observed relative intensity curves drawn as full lines in Fig. 2 were obtained by plotting these calculated values of $\log \frac{D_0}{D_x}$ for corresponding values of $\frac{\sin\theta}{\lambda}$. The observations made above concerning the characteristics of the diffraction patterns are still more obvious in these relative intensity curves.

IV. Interpretation of Intensity Curves

The method of interpretation of the intensity curves consists of assuming a possible atomic arrangement for the glasses. The assumed structure is then checked by calculating the intensity of scattering for various values of $\frac{\sin\theta}{\lambda}$ by means of an equation derived by Zernicke and Prins[9] for intensity of scattering in liquids. The general form of this equation is

$$I = \Sigma_m \Sigma_n f_m f_n \frac{\sin s r_{mn}}{s r_{mn}},$$

Where f_m and f_n = atomic scattering factors for m and n atoms, respectively.

$$s = \frac{4\pi \sin\theta}{\lambda}$$

r_{mn} = distance between m and n atoms.

According to this equation the intensity of scattering at any particular angle depends on the character and number of atoms present and the distances between their centers. From the chemical composition, the kinds and number of atoms are known, and it remains only to assume the proper distances in order to calculate the scattering. When an intensity curve calculated by this method matches the observed curve and if the assumed structure is in accordance with

[9] F. Zernicke and J. A. Prins, "The Bending of X-Rays in Liquids as an Effect of Molecular Arrangement," *Z. Physik*, **41**, 184 (1927).

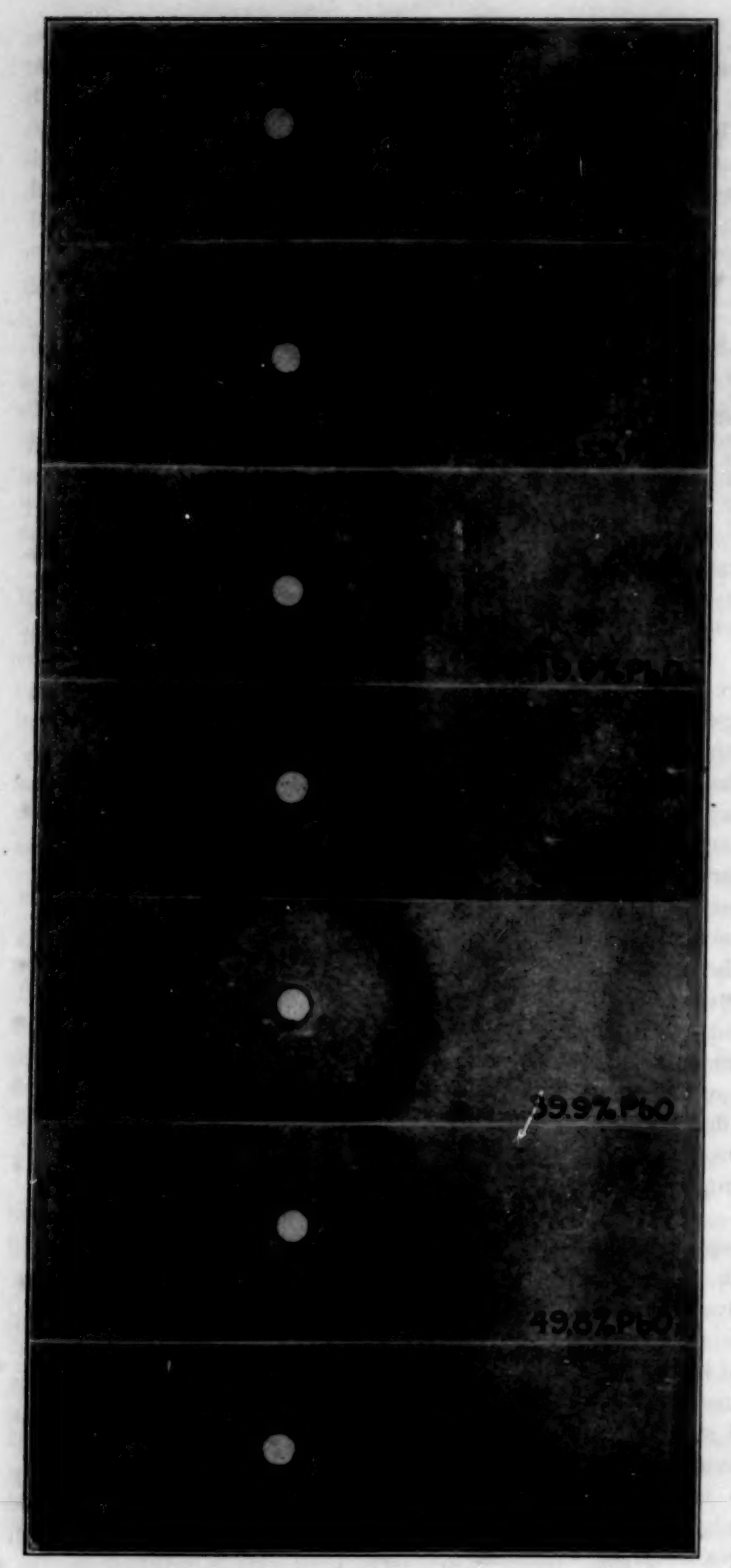

FIG. 1.—Diffraction patterns of lead oxide-silica glasses.

known atomic groupings of similar atoms in crystalline lattices, it can be concluded with some degree of certainty that the structure assumed is reasonably close to the actual arrangement.

The interpretation of the diffraction patterns for the lead oxide-silica glasses was simplified considerably by a study of the results obtained by Warren[8] and his assistants for the other glasses investigated. The apparent difference between the character of the diffraction patterns obtained in the present investigation and that for the soda-silica series was quite misleading, however, and their true similarity was not discovered until after several other possible atomic arrangements were tried.

The intensity curves obtained for the lead oxide-silica glasses give only one major peak which progressively moves to larger angles and sharpens considerably as the lead-oxide content increases. The curves for the series of soda-silica glasses gave two distinct major peaks. The height of the first peak decreased and the second one increased as the soda content increased, but there was no shift in the position of the bands. The reason for these differences at first appeared quite obvious. The intensity of scattering is proportional to the product of the atomic scattering factors, and since the atomic scattering factor for Pb^{++} is about eight times that for O^{--} or Si^{++++} those quantities in the summation which contained f_{Pb} and especially those containing $(f_{Pb})^2$ would overshadow all other effects. Furthermore, since the peak moves continuously to larger angles with an increase in lead-oxide content, it was interpreted to mean that the distances which were chiefly responsible for this main peak must be getting closer together. The only distance which might be expected to change continuously with an increase in lead-oxide content would be the Pb–Pb

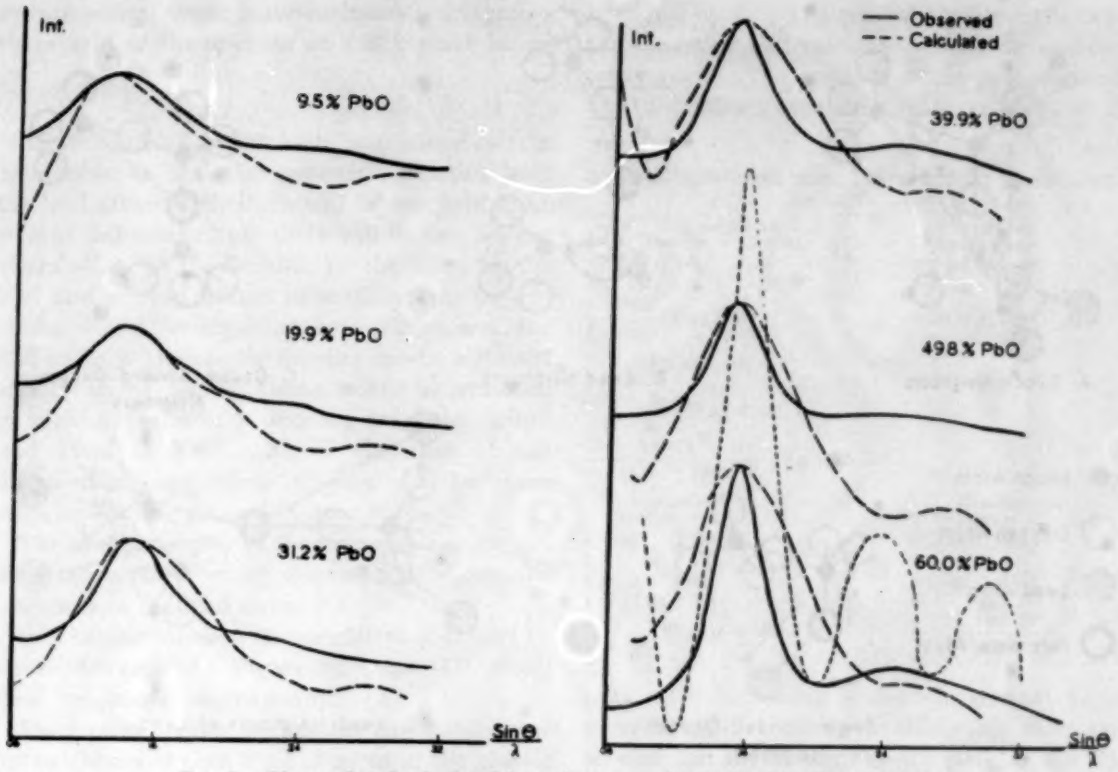

FIG. 2.—Observed and calculated intensity curves for lead oxide-silica glasses.

distance. On this basis, attempts were made to build up a structure fulfilling these requirements. Models of various types were constructed, but no arrangement could be derived which would give a constant Pb–Pb distance of the required magnitude.

Having failed in this approach to the solution of the structure, it was decided to ignore temporarily the preponderance of scattering which must arise from the Pb atom and start with new assumptions which were known to exist with some degree of certainty. These assumptions are (1) that the silicon atom will be surrounded tetrahedrally by four oxygens, (2) that some of these oxygens will be joined to other silicons and some to lead atoms, and (3) that each lead atom will be joined to at least two oxygen atoms. From these assumptions and with a knowledge of ionic radii, distances can be calculated between atoms. The number of each kind of atom about any one atom can be calculated from the chemical composition and the radius ratio.

The calculation of the general equation for the scattering of X-rays by a series of lead oxide-silica glasses follows the same procedure as that used by Warren and Loring[8] for soda-silica glasses. The composition of the glasses can be represented by $(PbO)_x\ (SiO_2)_{1-x}$, where x is the mol fraction of PbO. Then the relative numbers of the different atoms are (x)Pb, $(1 - x)$ Si, and $(2 - x)$O. The oxygens must be placed into two classes; those shared by silicon atoms, called O_2, and those bonded to only one silicon, called O_1. If γ is the fraction of oxygens bonded to only one silicon, then $O_1 = \gamma O$ and $O_2 = (1 - \gamma)O$. For each O_1 there is one silicon-oxygen bond, and for each O_2 there are two silicon-oxygen bonds. Since the total number of silicon-oxygen bonds must be equal to four times the number of silicon atoms,

$$1\,O_1 + 2\,O_2 = 4Si$$

or

$$\gamma O + 2(1 - \gamma)O = 4Si$$

$$2 - \gamma = 4\frac{Si}{O} = 4\frac{1 - x}{2 - x}$$

$$\gamma = 2 - 4\frac{1 - x}{2 - x}$$

$$\gamma = \frac{2x}{2 - x}.$$

The neighbors for the different atoms can now be calculated. The neighbors of any silicon atoms

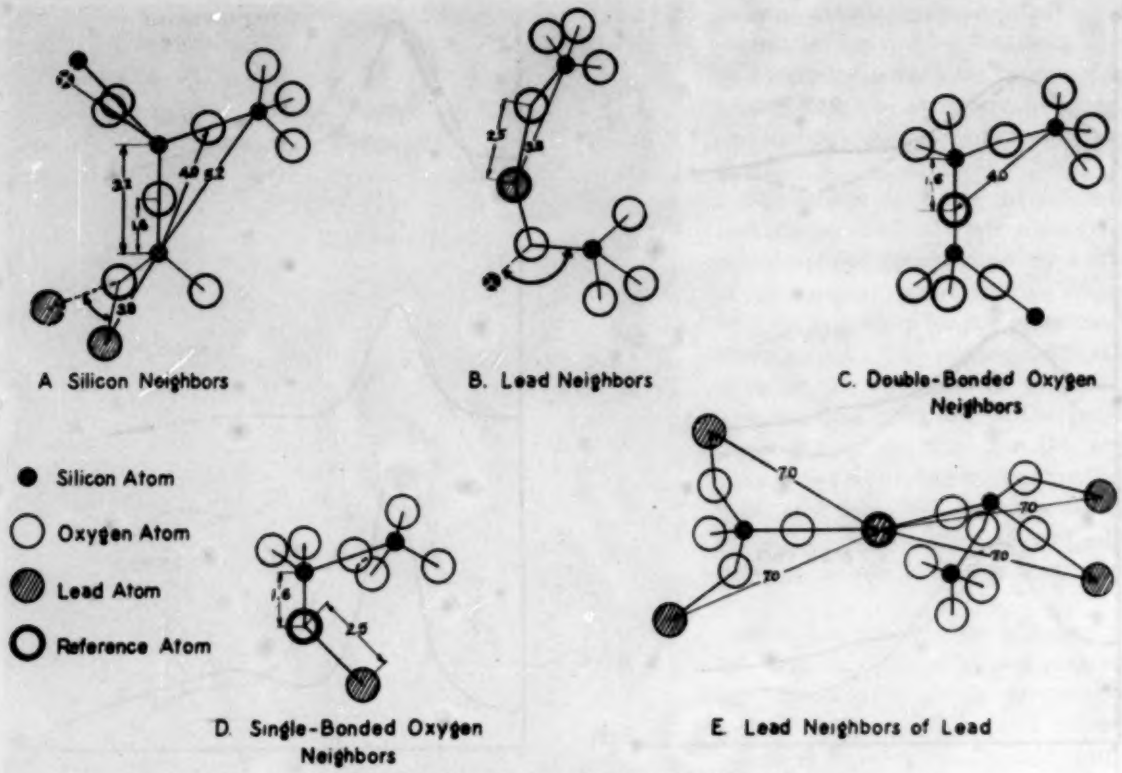

FIG. 3.—Atomic neighbors of lead oxide-silica glasses.

are as follows: first, the atom itself at zero distance. At a distance of 1.6Å there will be four oxygens. Of these oxygens, some will be attached to other silicon atoms and others to lead atoms. The total number of oxygen-silicon bonds is $\gamma O + 2(1-\gamma)O = (2-\gamma)O$. The fraction leading to O_2 is then

$$\frac{2(1-\gamma)O}{(2-\gamma)O} = \frac{2\left(1-\frac{2x}{2-x}\right)}{2-\frac{2x}{2-x}} = \frac{2-3x}{2(1-x)}.$$

Therefore, of the four oxygens surrounding the original silicon

$$4 \cdot \frac{2-3x}{2(1-x)} = \frac{4-6x}{1-x}$$

will be joined to other silicons at 3.20Å and

$$4\left(1-\frac{2-3x}{2(1-x)}\right) = \frac{2x}{1-x}$$

will be attached to lead atoms at 3.80Å.[10] Each of the $\frac{4-6x}{1-x}$ silicons will be surrounded by four oxygen atoms one of which has already been counted. There will be then $3\left(\frac{4-6x}{1-x}\right)$ oxygens at 4.00Å. Again there will be $\frac{2-3x}{2(1-x)}$ times this number of oxygens or

$$\frac{2-3x}{2(1-x)} \cdot 3\left(\frac{4-6x}{1-x}\right) = \frac{(3-5x)(4-6x)}{(1-x)^2}$$

which will be bonded to other silicons at 5.20Å from the original atom. It is apparent from Fig. 3(A) that this is as far away from the original atom as definite distances can be assigned. The next three oxygens will vary in their distance from the initial atom, depending upon how the structure is twisted. Following a practice used by Warren this last silicon and one-half of each of its surrounding oxygens may be considered an SiO_2 group. Therefore, the last calculated silicons at 5.20Å can be replaced by $\frac{(3-5x)(4-6x)}{(1-x)^2}$ SiO_2 groups at 5.20Å. And since one-half of

[10] If the Si–O–Pb angle is 180°, the Pb–Si distance would be 4.1Å. In the distorted glass network, this angle is probably never 180°, and the true Pb–Si distance is something less than 4.1. A value of 3.80Å gave the best results in the calculated curves.

the preceding oxygen is also included in this group, the weight of the oxygens at 4.00Å must be reduced to $3\left(\frac{2-3x}{1-x}\right)$.

The neighbors about each lead atom can be determined in a similar manner. Starting with the lead atom itself, there will be one lead atom at zero distance. Next there will be two oxygen atoms at 2.50Å.[11] Because of the sizes of the lead and oxygen atoms more than two oxygen atoms would be expected to pack around the lead atom. The proper number can be obtained only by trial. Silicon atoms would be attached to these oxygens at a distance from the initial lead atom of 3.8Å. As in the case of the silicon neighbors, these silicons can be more properly considered as SiO_2 groups.

The proper number of these groups can be determined only by trial. Figure 3(B) shows the neighbors of the lead atom.

In a similar manner, the neighbors of O_1 and O_2 can be determined. Figure 3(C), and (D), shows these neighbors schematically.

The final values for the number and distances of the neighbors of each kind of atom in the glasses which gave the best match with the intensity curves are summarized in Table II.

By substituting the values of Table II in the general equation, the following working equation was obtained:

$$I = (1-x)f_{Si}\left\{f_{Si} + 4f_O\frac{\sin 1.60s}{1.60s} + \frac{4-6x}{1-x}f_{Si}\frac{\sin 3.20s}{3.20s} + \frac{2x}{1-x}f_{Pb}\frac{\sin 3.80s}{3.80s} + \frac{3(2-3x)}{1-x}f_O\frac{\sin 4.00s}{4.00s} + \frac{(3-5x)(4-6x)}{(1-x)^2}f_{SiO_2}\frac{\sin 5.20s}{5.20s}\right\} + xf_{Pb}\left\{f_{Pb} + 3f_O\frac{\sin 2.50s}{2.50s} + 6f_{SiO_2}\frac{\sin 3.80s}{3.80s}\right\} + 2xf_O\left\{f_O + f_{Pb}\frac{\sin 2.50s}{2.50s} + f_{Si}\frac{\sin 1.60s}{1.60s} + 1.5f_O\frac{\sin 2.65s}{2.65s} + \frac{3-5x}{1-x}f_{SiO_2}\frac{\sin 4.00s}{4.00s}\right\} + (2-3x)f_O\left\{f_O + 2f_{Si}\frac{\sin 1.60s}{1.60s} + 3f_O\frac{\sin 2.65s}{2.65s} + \frac{6-16x}{1-x}f_{SiO_2}\frac{\sin 4.00s}{4.00s}\right\} + \text{continuous.}$$

The value of the intensity of scattered radiation was calculated for each value of $\frac{\sin\theta}{\lambda}$ for all the glasses.[12] The results are plotted in dotted lines in Fig. 2. The general agreement between the observed and calculated intensity curves is fairly good. The position of the peaks coincide and a tendency toward the sharpening of the peak with increasing lead-oxide content is obvious in both. There is considerable difference, however, in the breadth of the peak at the high lead-oxide end of the series, and the second diffused peak occurs at a slightly smaller angle than calculated. Another maximum at a small angle is also indicated in the calculated curve but is absent in the observed curve.

TABLE II

ATOMIC NEIGHBORS AND DISTANCES IN LEAD OXIDE-SILICA GLASSES

Si neighbors (weight = $(1-x)$)	Pb neighbors (weight = x)
1 Si at 0.00	1 Pb at 0.00
4 O at 1.60	3 O at 2.50
$\frac{4-6x}{1-x}$ Si at 3.20	6 SiO_2 at 3.80
$\frac{2x}{1-x}$ Pb at 3.80	
$\frac{3(2-3x)}{1-x}$ O at 4.00	
$\frac{(3-5x)(4-6x)}{(1-x)^2}$ SiO_2 at 5.20	

O_1 neighbors (weight = $2x$)	O_2 neighbors (weight = $(2-3x)$)
1 O at 0.00	1 O at 0.00
1 Pb at 2.50	2 Si at 1.60
1 Si at 1.60	3 O at 2.65
1.5 O at 2.65	$\frac{6-10x}{1-x}$ SiO_2 at 4.00
$\frac{3-5x}{1-x}$ SiO_2 at 4.00	

Several factors can be noted which might be responsible for the discrepancies between the calculated and observed intensity curves. In the first place, the neighbors about any lead atom were considered out only to a distance of 3.80Å and these included no Pb–Pb distances. It can be calculated from the density data that the average Pb–Pb distance would vary with composition from about 4.00 to 6.50Å. In the final intensity-

[11] The Pb–O distance in crystalline PbO is 2.33Å. (J. A. Darbyshire, "An X-Ray Examination of the Oxides of Lead," *Jour. Soc. Chem.* [London], pp. 211–19 (1932)). In this structure the Pb atom is surrounded with O on one side only. The Pb–O distance, when the Pb atom is completely surrounded by oxygen, will be something greater; 2.50Å was considered to be a reasonable figure.

[12] The scattering factors given by Linus Pauling and J. Sherman ("Screening Constants for Many-Electron Atoms: the Calculation and Interpretation of X-Ray Term Values and the Calculation of Atomic Scattering Factors," *Z. Krist.*, **81** [1–2] 1–29 (1932)) were used in this calculation. The values of f_{SiO_2} were calculated from $f_{SiO_2} = f_{Si} + 2f_O\frac{\sin 1.60s}{1.60s}$

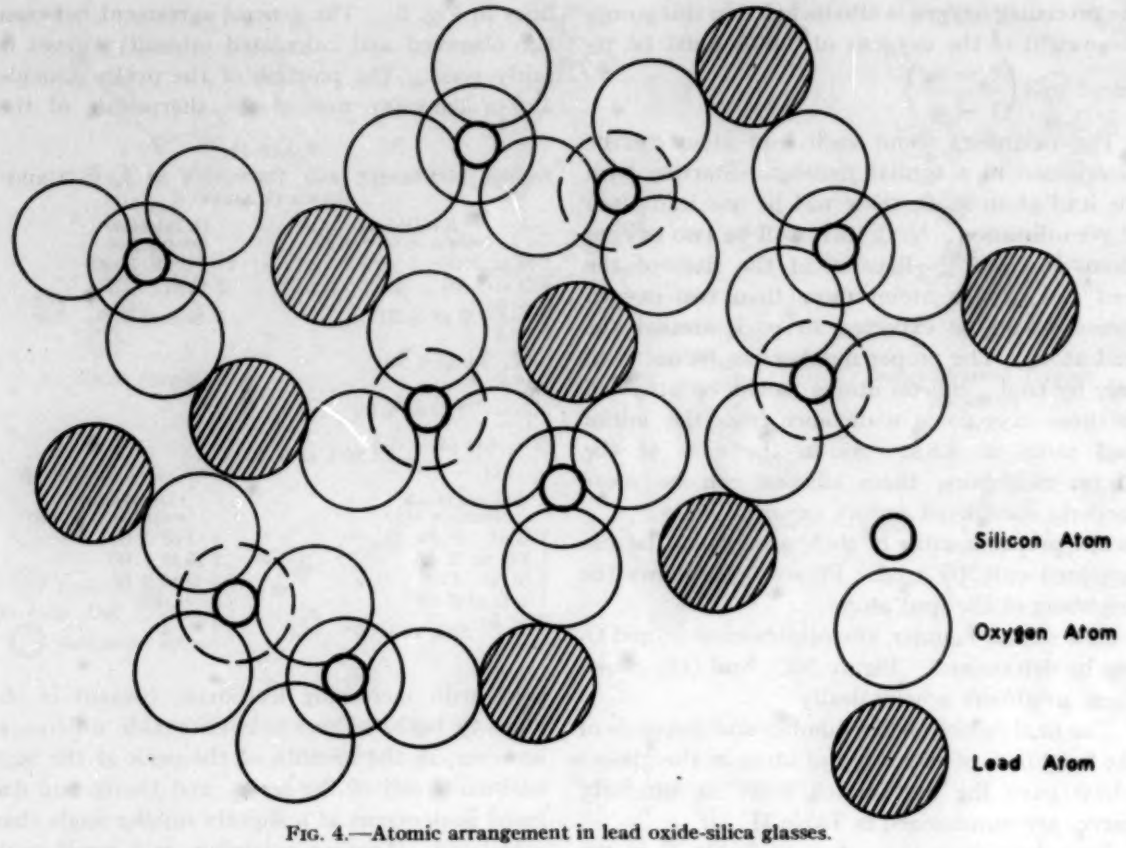

FIG. 4.—Atomic arrangement in lead oxide-silica glasses.

curve calculation, it was assumed that these distances were not sufficiently constant to give discrete scattering, but if only a small fraction of the lead atoms occurs at a specific distance their effect will be large and they will appreciably influence the resultant scattering. An example of the effect of Pb–Pb distances is illustrated by a calculation made on the glass containing 60 mol % of lead oxide. A part of the assumed structure is shown in Fig. 3(E). If it is assumed that the Pb–O–Si angle is 180°, a definite Pb–Pb distance must occur having a value of about 7.00Å. In the glass containing 60% of lead oxide, there will be four lead neighbors to each lead atom at this distance. If a factor of this magnitude is added to the summation of the calculated intensity curve, it will have a shape represented by the light dashed line in Fig. 2. It can be seen that, by allowing for this Pb–Pb distance in full strength, it has changed the calculated curve too far in the opposite direction. Such a Pb–Pb distance would not be expected to be sufficiently constant for discrete scattering, and yet if an atom group were favorably situated these distances would be constant. Because it is impossible to evaluate the number which might occur, the entire term has been omitted.

Another factor which might affect the correlation of the calculated with the observed curves is the continuous scattering at large distances. This was omitted in the summation. Probably of greater importance, however, are the discrete distances which occur between those evaluated and a distance beyond which a perfectly continuous distribution of scattering matter does exist. This omission is especially critical at small angles and may be responsible for the rise in calculated scattering at $\frac{\sin\theta}{\lambda} = 0.08$.

Considering, then, these factors which can not be evaluated, it can be concluded that the calculated curves differ from the experimental ones in degree only and that an atomic arrangement similar to that derived would give a diffraction pattern like those obtained for the lead oxide-silica glasses.

It may thus be inferred that the structure of lead oxide-silica glass probably consists of an

interlocking network of atoms in which (*a*) each silicon is surrounded by four oxygens, (*b*) the oxygens lie between two silicon atoms or a silicon and a lead atom, and (*c*) the lead atoms are always separated by silicon-oxygen tetrahedra and never occur adjacent to each other or joined to each other through a common oxygen. Figure 4 is a schematic diagram of this structure of lead oxide-silica glasses.

The structure of lead oxide-silica glasses thus derived is almost identical with that obtained for soda-silica glasses by Warren and Loring.[8c] It is only necessary to change their picture by replacing two sodium atoms with a lead atom. This further substantiates the accuracy of the method and the correctness of the assumptions upon which the structure was based. Two sets of diffraction patterns of apparently different type are matched with calculated intensity curves by applying the same reasoning to each. The differences in the patterns are due only to differences in atomic radii and scattering power and not to any fundamental difference in the character of the bonding in the glass.

V. Summary of the Atomic Arrangement in Lead Oxide-Silica Glasses

The X-ray analysis of the structure of lead oxide-silica glasses indicates that the glasses are composed of a continuous, randomly oriented network of atoms. The diffraction patterns for the series of glasses show a continuous change as lead oxide is added. There is no indication of crystallite formation or sudden change in atomic arrangement with change in composition. The results of this study substantiate the structure derived by Warren and Loring[8c] for soda-silica glasses and is in accord with Zachariasen's[5] picture of the glassy state.

II. THE CORRELATION OF PHYSICAL PROPERTIES WITH ATOMIC ARRANGEMENT

By George J. Bair

Abstract

Physical properties of lead oxide-silica glasses were studied in an attempt to correlate these properties and explain the anomalies occurring in their temperature and time curves on the basis of the structure of glass as derived in Part I of this investigation. The transformation point is defined as a critical temperature, and an explanation is offered for the dependence of this temperature on heat treatment and conditions of test. The change in the apparent viscosity of a glass fiber with time is explained on the basis of the distorted glass structure. The Adams and Williamson equation for strain release is adapted to substantiate the theory of the structure of glass consisting of a continuous, randomly oriented, three-dimensional network of atoms.

I. Introduction

At the inception of this investigation, it was decided to study a series of lead oxide-silica glasses (1) by means of the X-ray to derive a probable atomic arrangement and (2) by examining the physical properties of these glasses in relation to their structure. The results of the X-ray study have been described.[1] The derived structure of the series of lead oxide-silica glasses can best be described as a continuous, randomly oriented, three-dimensional network of silicon-oxygen tetrahedrons and lead atoms. No indication of discrete aggregates or molecules was found.

Most of the discussions of the glassy state, based on the interpretation of physical property data, concern the so-called "transformation point." Thermal expansion, electrical conductivity, index of refraction, and many other properties show sudden discontinuities in their temperature curves in the upper annealing range. This phenomenon has been pointed out by many authors and is discussed at length by Berger,[2] who concludes that the temperature of this break is the transformation point between brittle glass and a viscous liquid.

In Littleton's[3] discussion of this critical tem-

[1] See Part I, "Atomic Arrangement," p. 339.

[2] See footnote 4, p. 339.

[3] See footnote 3(*a*), p. 339.

perature, several anomalies are shown. The strongest evidence against the transformation point, apparently, is the absence of any indication of a discontinuity in the viscosity-temperature curve in the upper annealing range. Another fact which seems contradictory to a critical temperature is the apparent shift of the transformation point with heat treatment and test procedure.

Lillie's[4] work on the viscosity of glass in the annealing range has had much influence on recent studies on the nature of glass. He found that an equilibrium rate of elongation of a glass fiber was approached from different directions when the specimen was given a previous heat treatment above and below the test temperature. He assumes that the change in the rate of elongation with time is due to a change in the viscosity of the glass with time, and he interprets this to indicate that there is an equilibrium degree of aggregation or association for each temperature. This view has been quite generally accepted in this country.

In this second part of the study of the constitution of glass, it seemed desirable (*a*) to obtain data on the transformation points of the series of lead oxide-silica glasses and to attempt to explain the results on the basis of their atomic arrangement, and (*b*) to study the viscosity-time relations of glass fibers and to justify the results, if possible, on the basis of a structure which possesses no discrete aggregates or molecules.

Thermal expansion, heating and cooling curves, and the viscosity of four of the series of lead oxide-silica glasses were studied. The preparation and analysis of the glasses were described in Part I of this investigation.[1] The molar compositions of the glasses used are given in Table I.

TABLE I

MOLAR COMPOSITION OF GLASSES

Glass No.	SiO_2	PbO	Al_2O_3 and Fe_2O_3
30	68.4	31.2	0.4
40	59.8	39.9	.3
50	49.8	49.8	.4
60	39.4	60.0	.6

II. Thermal Expansion

The first measurements of thermal expansion in this investigation were made with a Fizeau interferometer. Three rods of the glass, one centimeter long and about one millimeter in diameter, were clamped vertically to a brass ring.

[4] H. R. Lillie, "Viscosity-Time-Temperature Relations in Glass at Annealing Temperatures," *Jour. Amer. Ceram. Soc.*, **16** [12] 619–31 (1933).

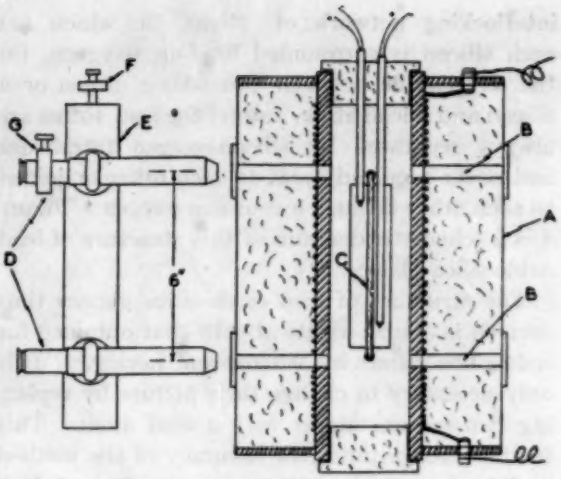

FIG. 1.—Thermal expansion apparatus (top view).

When this assembly was placed on a quartz disk and a second quartz disk was placed on top, the small rods were supporting considerable load. In the first run, it was found that the weight on the test specimens was so great that they distorted below the transformation temperature and accurate readings in that range were impossible.

(1) Expansion Apparatus

A method which imposes no load on the specimen was finally adopted for the study. A plan view of the apparatus is shown in Fig. 1. The apparatus consisted of a horizontal tube furnace, *A*, with two slots, *B*, in the tube wall so that the beaded ends of a 6-inch specimen, *C*, could be viewed through a pair of parallel telescopes, *D*. The telescopes were rigidly mounted on a slide, *E*, which could be shifted by means of a fine screw, *F*. One telescope was equipped with a filar micrometer eyepiece, *G*, so that, after setting the crosshair of the other telescope tangent to one end of the fiber, the change in length of the fiber could be determined by the micrometer. The micrometer used measured changes in length to within 0.002 millimeter. The rate of heating of the furnace was manually controlled by means of a transformer-type voltage regulator and was maintained at about 3°C per minute in all tests. The temperature of the specimen was measured with two chromel-alumel thermocouples placed so that their hot junctions were one inch from each end and very close to the test piece. The temperature variations along the test rods as read by these two thermocouples were less than 2°C throughout the tests.

(2) Results of Expansion Tests

Fresh drawn, unannealed fibers were used in the furnace and test runs were repeated on them until no break was found in the curve below the critical temperature. Figure 2 shows the expansion-temperature curves for glass No. 40. During the first run (curve 1), the fiber expanded uniformly up to about 300° at which temperature it started to contract owing to the release of the tensile strain placed in the rod during drawing as well as to the possible adjustment of bond distortion resulting from rapid cooling. This point of inflection corresponds to the lower limit of the annealing range. The heating of the fiber was continued to a temperature below the softening point. The specimen was then cooled slowly in the furnace. The second and third runs (curves 2 and 3) on the same fiber still showed signs of annealing but with decreasing amounts. Curve 3 is nearly a straight line; curves 4 and 5 give the expansion curves for another fiber of glass No. 40. The similarity of the curves gives evidence of the reproducibility of the results and the constancy of the critical temperature.

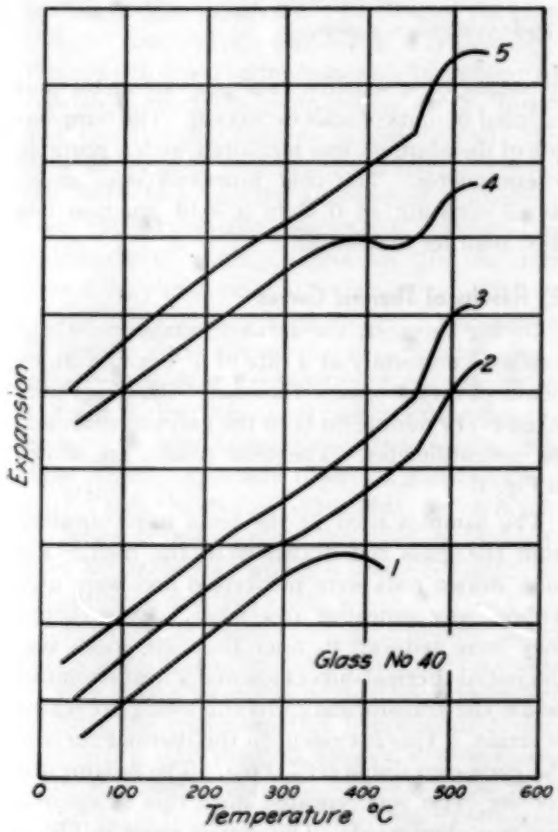

FIG. 2.—Thermal expansion with various heat treatment.

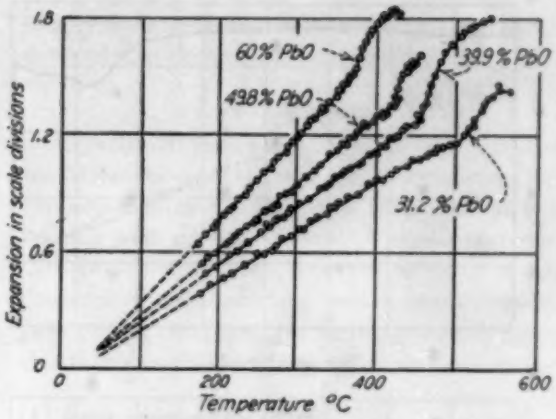

FIG. 3.—Expansion of lead oxide-silica glasses.

Figure 3 shows the final expansion-temperature curves for glasses Nos. 30, 40, 50, and 60. A definite break was obtained in each curve. The temperature of this break, which has been defined as the transformation point or the critical temperature, changes continuously with concentration and occurs several hundred degrees below the liquidus temperature. The transformation points are given in Table II. The values of coefficients of expansion, calculated from the slopes of the expansion curves within the temperature range 250° to 350°C, are also included in this table.

At a temperature of about 50° above the critical temperature, the glass became so soft that the protruding end of the glass fiber could be seen to sag slowly under its own weight. A slight load, such as would be obtained from an Ames dial, would cause this distortion to take place at a still lower temperature and would indicate a shorter softening range. For the same reason a load on the fiber may change the slope of the curve above the critical temperature and even below this temperature in a smaller degree.

TABLE II

TRANSFORMATION POINTS AND COEFFICIENTS OF EXPANSION (250° TO 350°C) OF LEAD OXIDE-SILICA GLASSES

PbO content (mol %)	Transformation temperature (°C)	Coefficient of expansion (cm./cm./°C)
31.2	506 ± 5	6.51×10^{-6}
39.9	460	7.74
49.8	420	9.16
60.0	350	11.20

III. Thermal Curves

Heating and cooling curves were obtained on the lead oxide-silica glasses in an attempt to throw further light on the nature of the change

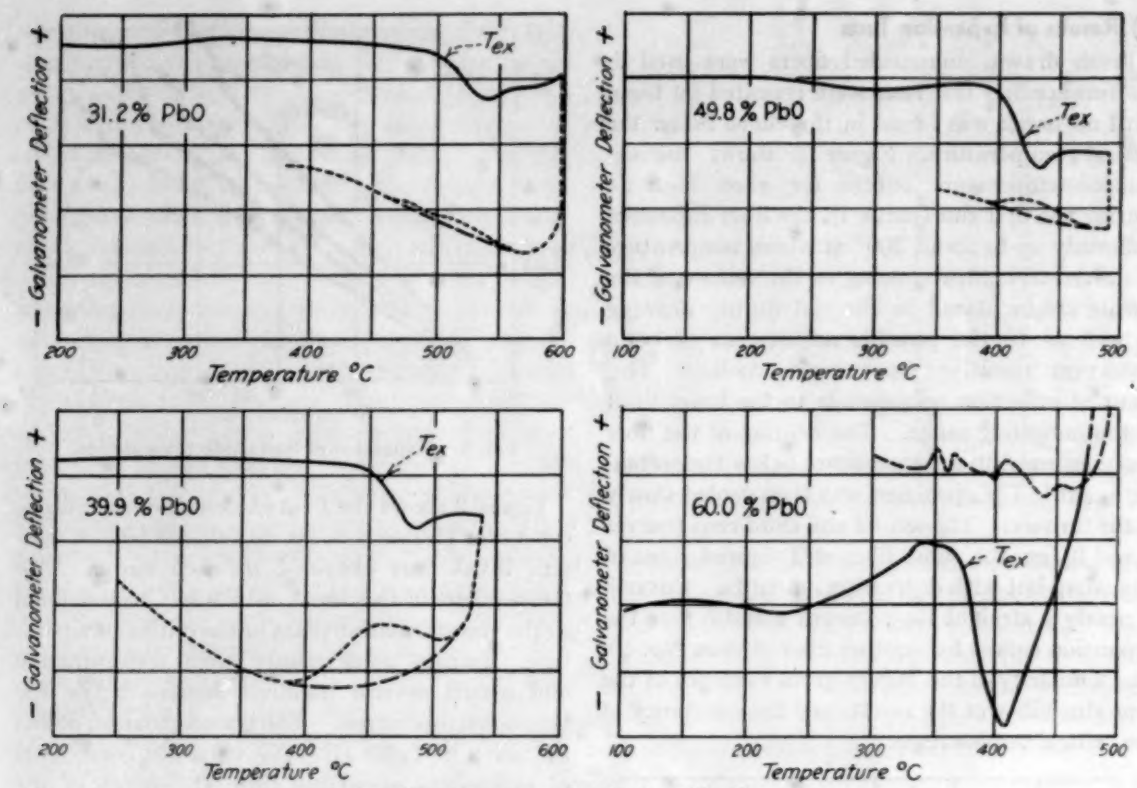

FIG. 4.—Thermal curves for lead oxide-silica glasses.

which occurs at the temperature of the break in the expansion curve. The method used in obtaining these curves was essentially the same as that used by Tool and Eichlin.[5]

(1) Experimental Procedure

The sample of glass, which had been ground in an agate mortar to pass a 100-mesh sieve, was placed in the one side of a double crucible. The heating and cooling rate of the glass was compared with that of granular, electrically fused alumina placed in the other half of the crucible. The hot junctions of a chromel-alumel differential thermocouple were imbedded in the centers of the two materials.

The actual temperature-difference between the sample and the reference material depends on several factors. The relative conductivity of the materials, the degree of packing, the specific heat, the size of the sample, and the rate of heating influence the apparent magnitude of the heat effect. Because of these factors, no attempt was made to determine the actual temperature-difference. The relative difference was measured by means of a sensitive wall galvanometer and recorded in units of scale deflection. The temperature of the alumina was measured with a portable potentiometer. The cold junctions were maintained constant at 0°C in a cold junction box filled with ice and water.

(2) Results of Thermal Curves

During the test, the furnace temperature was increased uniformly at a rate of 6° per minute by means of an automatic rheostat. Readings were taken every minute on both the galvanometer and the potentiometer. The final results are shown in Fig. 4.

The samples used in the tests were obtained from the glass rod drawn from the melt. The fresh drawn rods were pulverized and were used without any annealing treatment. Even though they were reduced to finer than 100-mesh size, the initial thermal curves showed a heat evolution below the transformation point owing to release of strain. This is evident in the thermal curve of the glass containing 60% PbO. The heating and cooling cycle was repeated until this exothermal rise was eliminated. The curves given in Fig. 4,

[5] See footnote 2(*b*), p. 339.

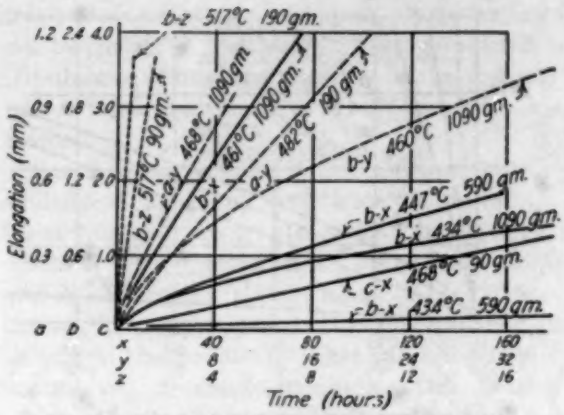

FIG. 5.—Elongation-time curves for glass No. 30.

except that for glass No. 60, are the result of the second or third run on the same sample. In the case of glass No. 60, devitrification took place so rapidly above the transformation point that a second run was not comparable.

The cooling curves were obtained by opening the furnace switch and allowing the furnace to cool by radiation. As a result, a maximum rate of cooling was rapidly approached, followed by a gradually decreasing rate. Because of the higher thermal conductivity and lower heat capacity of the glasses, the thermocouple in the glass more nearly followed the temperature of the furnace and that in the alumina lagged behind. This gave a negative temperature-difference between the two which rapidly approached a maximum and then gradually decreased. Under these conditions of cooling, however, the difference should vary uniformly and the curve, in exclusion of any thermal effects, should be a smooth line. In Fig. 4, where the actual cooling curves represented by a short-dashed line deviate from this uniform cooling, the ideal cooling curve has been drawn in a long-dashed line. It is obvious from these curves, especially those for glasses Nos. 40 and 50, that the thermal effect obtained on heating the glass is reversible. The cooling curve for glass No. 60 is very irregular and gives indication of continued devitrification.

This thermal method of determining critical points is qualitative in character. It is impossible by this means to assign a particular value definitely to the transformation temperature because during the test there is always a temperature gradient through the sample itself. The transformation points determined by means of expansion measurements are indicated on Fig. 4 as T_{ex}. These show the correlation of the two methods in determining the critical temperature.

IV. Viscosity

A satisfactory explanation of the apparent transformation point must also be consistent with properties other than thermal expansion and the heating and cooling curves. Viscosity-temperature curves have been reported to show no discontinuity corresponding to a transformation point. To complete the present investigation, this fact was checked for lead oxide-silica glasses.

(1) Experimental Procedure

The method used to measure the viscosity of the glasses was similar to that used by Lillie.[6] A glass fiber, about 1 millimeter in diameter and 250 millimeters long, after being accurately calipered, was suspended vertically in a furnace. When temperature equilibrium was obtained, a load was hung on to the lower end of the test specimen and its change in length was periodically measured.

The apparatus used was described by Norton.[7] The furnace consisted of a stainless-steel tube wound with nichrome wire so that the center section and the two ends could be regulated separately. The furnace control was operated by the expansion of the furnace tube. The expansion of this metal tube was magnified fifty times by a lever arm which opened and closed contacts, disconnecting and connecting a large resistance in parallel with the main, current-regulating rheostat. The current was thus made

[6] H. R. Lillie, "Viscosity of Glass between the Strain Point and Melting Temperature," *Jour. Amer. Ceram. Soc.*, **14** [7] 502–11 (1931).

[7] F. H. Norton, "Measuring the Viscosity of Glass," *Glass Ind.*, **16** [5] 143–44 (1935); *Ceram. Abs.*, **14** [7] 160 (1935).

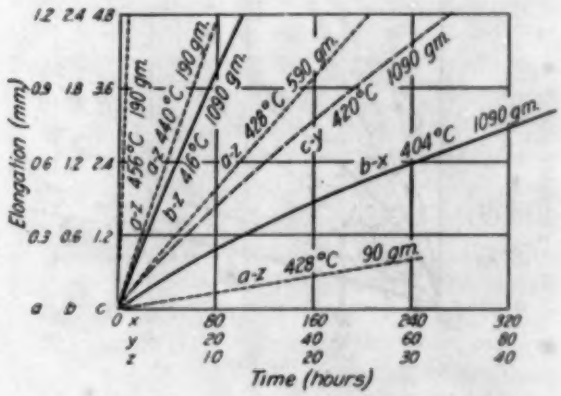

FIG. 6.—Elongation-time curves for glass No. 40.

to fluctuate between two values, making a complete cycle about three times a minute. With these rapid fluctuations and the damping effect of the heavy steel furnace tube, it was impossible to detect corresponding temperature fluctuations by means of a type-K potentiometer and three chromel-alumel thermocouples placed along the glass specimen. With this control, the temperature variation over periods of weeks was less than 0.5°C.

The test specimen was beaded at the ends and fitted into a socket in the stainless-steel hanger suspended from a rigid mounting at the top of the furnace. A lower hanger upon which the weight was hung was similarly suspended from the lower end of the test fiber. The extension of the specimen under test was measured by means of a telescope mounted on a micrometer slide and sighted on a hairline mark on the lower hanger. Changes of length could be accurately measured to 0.002 millimeter. Since all runs were made at a constant temperature, there was no correction necessary for the expansion of the metal hangers, and under the light loads and low temperatures any "creep" of the stainless steel hanger rods could be neglected. Readings were taken as often as significant changes could be obtained. The time between readings ranged from one minute, when flow rates were high, to a day at the lower temperatures. The tests were continued until an apparent constant rate of elongation was obtained. In many of the tests after constant flow was obtained, the load was removed and readings were continued to measure the delayed elastic contraction, and in some tests repeated loadings and unloadings were made.

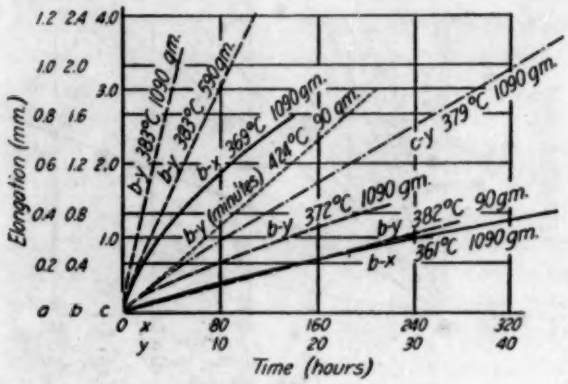

FIG. 7.—Elongation-time curves for glass No. 50.

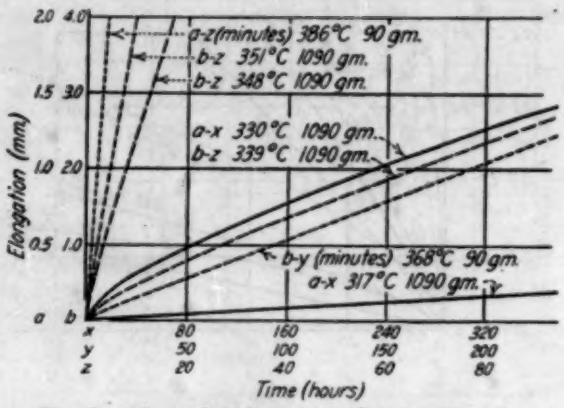

FIG. 8.—Elongation-time curves for glass No. 60.

(2) Results of Fiber Elongation Tests

The data obtained in the fiber elongation tests are plotted in Figs. 5 to 8. The curves for each glass are plotted on separate graphs. These curves are not directly comparable because they are plotted on different scales and were obtained with different loads. In all cases, after a freshly drawn fiber was loaded, it showed a decreasing rate of flow with time. This checked Lillie's findings for soda-lime glass. The fiber seemed to approach some equilibrium condition of flow. This final slope, after steady flow was reached, was substituted in Lillie's equation for viscosity:

$$\eta = \frac{60\, lmg}{3\pi r^4 S}$$

Where η = viscosity (poises)
l = length of fiber (cm.)
m = total suspended weight (grams)
g = gravity constant
r = radius of fiber (cm.)
S = slope of elongation curve (cm./min.).

The logarithm of these final calculated apparent viscosities are plotted against temperature in Fig. 9. There are no discontinuities in these curves corresponding to a transformation point. This confirms Lillie's results.

V. Discussion of the Transformation Point

At this point it may be well to take inventory of what is now known concerning the transformation point. It has been shown in this investigation that a definite discontinuity occurs in the expansion and the heating and cooling curves at a definite and constant temperature for each glass when tested under a standardized procedure. The numerous examples in the literature, showing that the temperature of these discontinuities can be made to shift with treatment and methods of

test and in some cases disappear altogether, can not be ignored. Another fact to be considered is the absence of any discontinuity in the viscosity curves when measured by the fiber-elongation method.

Recalling the original assumption based on the evidence of the X-ray investigation, the lead oxide-silica glasses consist of a continuous, randomly oriented, three-dimensional network of lead atoms and silicon-oxygen tetrahedrons. The explanation of the transformation point must include an answer to the question, "What can happen to a continuous, distorted structure upon heating which will give the reversible discontinuity in the expansion and heating curves at a definite temperature, depending upon conditions of test, and

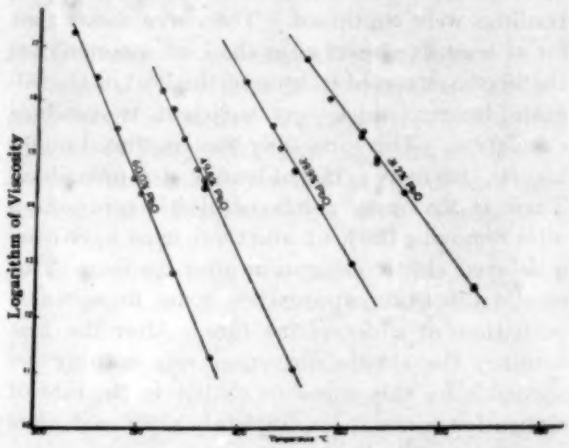

FIG. 9.—Viscosity-temperature curves for lead oxide-silica glasses.

show no evidence of this change in the fiber elongation tests?"

At room temperature the glass is a solid. The forces holding the atoms together in a glass must be essentially the same as those in a crystal. The only difference is the inherent distortion of atomic bonding in the randomly arranged network of a glass structure. This results in a higher energy content in a glass than in a crystal. The distorted bonds will require less additional energy to break them. At room temperature, the energy of thermal vibration is not sufficient to break bonds, but as the temperature is increased an energy level will eventually be reached where the most highly distorted bonds will break and the atoms affected will seek a more comfortable association.

In a rapidly cooled glass, atomic bonds must exist in a wide range of distortion. The release of this strain would then occur gradually as the glass is heated and would not be expected to give the sudden break exhibited at the transformation point. The effect would be one of energy release and might correspond to the exothermal effect produced in unannealed glass below the transformation point. The change which would occur in the glass structure would be irreversible and tend toward greater density. This may explain the gradual contraction in the unannealed glass below the transformation point. The transformation point can not be due to this annealing action.

Owing to the nature of the atomic units in glass-forming materials, the increase in orderliness of the atomic network can be carried only to a certain limit. In order to have a silica glass arrange its atoms in a perfectly orderly arrangement, that is, crystallize, the silicon-oxygen tetrahedrons must orient themselves. To do this, at least two of the oxygen atoms of a tetrahedron must break away from their associates at the same time and, with the same object in view, adjust themselves to new affections in just the right way to marshall the structure into a crystal. Devitrification occurs in some glasses at fairly low temperatures but, under the conditions by which a crystal must grow in a glass, it also must be a very slow process and could not be responsible for the sudden discontinuity in the property-temperature curves.

It is conceivable that the increase in orderliness of the structure of a glass as it is heated will approach a condition of the most orderly arrangement that a random structure can possess. This means that the bond distortions will approach rather constant values so that when the thermal energy reaches a certain level, several atoms throughout the structure will be able to break free from their surrounding forces at the same time, and, although the forces of attraction between atoms are acting continuously, the over-all most orderly arrangement possible to glass has been obtained and the new associations formed will be no more secure than the old. Bonds continue to make and break at a constant rate and, in effect, the glass becomes a liquid. Such an explanation seems the only logical one to produce a sudden break in the properties of a random structure as it is heated.

This theory of the transformation point assumes that the only forces acting on the atoms are those between the atoms themselves, which tend to bind

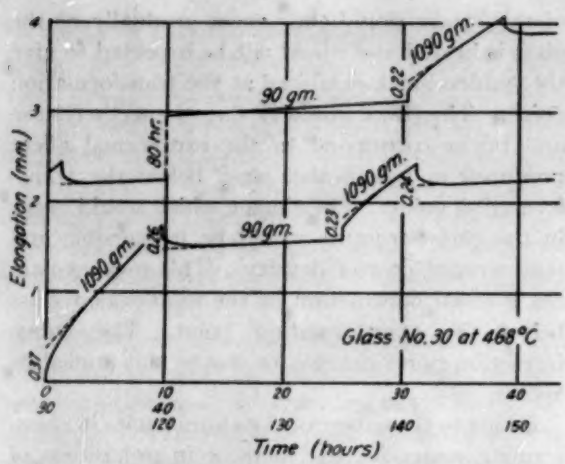

FIG. 10.—Elongation-time curve with repeated loading.

them together, and the disrupting force of thermal vibration. If an external disrupting force is superimposed upon this system, the increased distortion resulting will permit the atoms to break apart at a lower temperature and the apparent transformation point may be shifted or so smeared that it is not detectable. The fiber-elongation method of measuring viscosity continuously distorts the network of atoms so that bonds break continuously below the true transformation point and it is impossible by this method to detect a discontinuity where the solid glass spontaneously becomes a liquid. Similarly, all other methods of test which superimpose additional distorting force upon the structure, such as the pressure from an Ames dial in measuring expansion or the force of an electric field in conductivity measurement, will shift or destroy the discontinuity at the critical temperature. This shift will be greater when sufficient time is allowed during test to permit continuous distortion.

According to this theory, the transformation point is defined here as the critical temperature at which the distorted structure of a glass, when slowly heated in the absence of any external force, spontaneously breaks down and some of the constituent atoms suddenly become mobile. The solid glass becomes a liquid. This sudden loosening of the glass structure is reversible and, upon slow cooling through the transformation point, the evidence of the sudden loss of a degree of freedom of atomic motion is again exhibited by those tests which impose no external force on the structure.

This picture of the transformation point is in accord with the experimental facts resulting from property measurements and is consistent with the structure of glass as suggested by X-rays.

VI. The Apparent Change of Viscosity with Time

According to the picture of the structure of glass presented here, it is difficult to justify any equilibrium association in a glass which is different for each temperature below the transformation point, such as Lillie's work seems to indicate. In an attempt to explain this inconsistency, further study of the fiber elongation method of measuring viscosities was conducted.

The results of one experiment are shown in Fig. 10. A specimen of glass No. 30 was tested at 468°C and, after allowing the fiber to flow under load until nearly constant rate of elongation was attained, the added load was removed and readings were continued. The curve shows that for at least five hours after the load was removed the fiber contracted in spite of the load of the 90-gram hanger which was sufficient to produce elongation. This same fiber was continued under test for two more cycles of loading and unloading. There is obviously a delayed elastic contraction after removing the load, and there must have been a delayed elastic elongation after loading. This elastic distortion approaches some finite value with time at a decreasing rate. After the first loading, the elastic distortion was entirely responsible for this apparent change in the rate of elongation, since it is completely recovered upon the release of the load.

It seems reasonable to explain the change in rate of flow with the initial loading in a like manner. A freshly drawn, unannealed fiber possesses considerable strain[8] due to rapid cooling. This strain is randomly oriented. Upon loading the fiber at a temperature where flow may occur, the original strain will be removed in the direction of flow and increase the magnitude of the elongation. The final strain due to the load will be directed along the fiber. Upon the release of the load, this final directional strain will be removed by an

[8] The term "strain" is used throughout this discussion to mean "bond distortion." The view taken here is that when these bond distortions are oriented in the same direction (compression or tension) birefringence results and the strain is visible in polarized light. In a quenched fiber, however, where the glass is cooled so rapidly that no zones of compression and tension are formed and the fiber is optically free from strain, there may still be a high degree of bond distortion, randomly distributed in the fiber. This strain is essentially the same as that occurring in visibly strained glass and is likewise removed by annealing.

elastic contraction, but, of course, the state of initial strain in the freshly drawn fiber will not be recovered.

Considering the change in the rate of elongation with time to be caused by an approach to an equilibrium degree of strain or distortion with a given load, it appears that this condition of maximum strain is approached with a decreasing rate. The process is the reverse of an annealing process. At a particular temperature, a definite time is required to put strain into the glass, and the more strain present in the glass the longer it will take to increase it another increment. If this correlation to annealing is true, the same laws which govern the rate of release of strain during annealing should be applicable to this rate of putting strain into the glass.

(1) Derivation of Elongation-Time Equation

Figure 11(A) represents a typical elongation-time curve for the flow of a glass fiber. Assuming that the viscosity of a glass is constant, the elongation due to viscous flow would be a function of time only and the elongation-time curve would be a straight line having a slope, c, equal to the slope of the actual elongation curve when it reaches a state of steady flow. The dashed lines in Fig. 11(A) represent this viscous flow. The amount of elongation in excess of that for viscous flow is then proportional to the strain, f', due to the suspended load, or

$$f' = E - ct$$

Where f' = strain
E = total elongation at time, t
c = slope of elongation-time curve after steady flow state is obtained.

A plot of f' against time is represented by the dashed lines in Fig. 11(B). The solid line on the same graph represents the rate of release of strain according to the Adams and Williamson equation[9]:

$$\frac{-df}{dt} = Af^2 \text{ or } = (B/\eta) f^2 \qquad (1)$$

$$\text{Then } \frac{1}{f} - \frac{1}{f_0} = At$$

$$\frac{1}{f} = \frac{1 + Atf_0}{f_0}$$

$$= \frac{f_0}{1 + Atf_0}. \qquad (2)$$

If the change in the rate of elongation obeys the same laws as the rate of release of strain, the two curves given in Fig. 11(B) will be symmetrical and

$$f' = f_0 - f \text{ or } f = f_0 - f'.$$

Substituting in equation (2)

$$f' = f_0 - \frac{f_0}{1 + Af_0t}$$

$$f' = \frac{Af_0^2t}{1 + Af_0t}. \qquad (3)$$

If the above assumptions are correct, this equation is then the equation of the dashed line in Fig. 11(B).

This equation can be transposed to a more useful equation by letting

$$f' = E - ct.$$

$$\text{Then } E = \frac{Af_0^2t}{1 + Af_0} + ct.$$

$$\text{Let } \alpha = Af_0$$
$$\text{and } \beta = Af_0^2,$$

$$\text{The equation becomes } E = ct + \frac{\beta t}{1 + \alpha t}. \qquad (4)$$

Since equation (4) is based on the assumptions illustrated in Fig. 11(B), in which f' must be zero when t is zero, this equation is valid only when the test is started with the fiber in the condition of zero strain. This condition is probably never

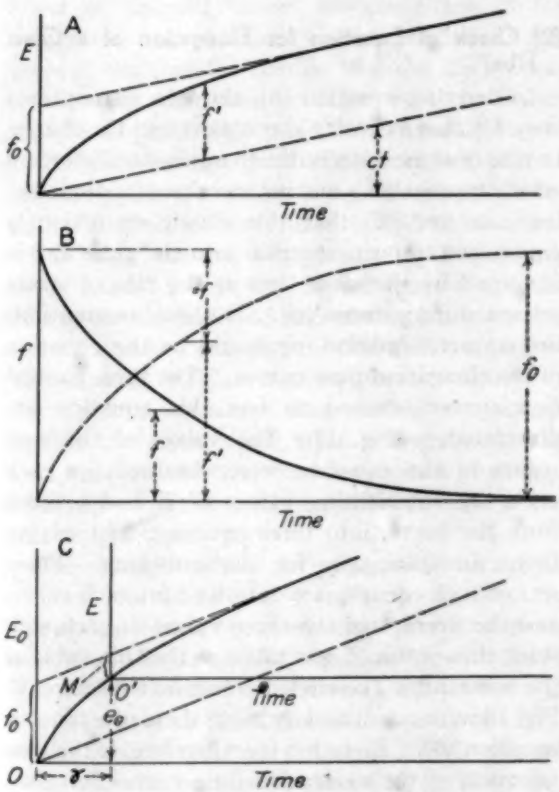

FIG. 11.—Viscosity-strain relation.

[9] L. H. Adams and E. D. Williamson, "Annealing of Glass," *Jour. Frank. Inst.*, **190**, 597 (1920).

obtained, and the test is always started from some position on the curve such as O' in Fig. 11(C). It is desirable then to make the equation general by transposing the origin to O'. To avoid confusion, equation (4) is rewritten with subscripts representing the conditions when the origin is at zero time and strain.

$$E_0 = ct_0 + \frac{\beta t_0}{1 + \alpha t_0}. \qquad (4^1)$$

The new origin has the coördinates

$$t = \gamma \text{ and } E = e_0.$$

Then $E_0 = E + e_0$
and $t_0 = t + \gamma.$

$$E + e_0 = ct + c\gamma + \frac{\beta t + \beta\gamma}{1 + \alpha t + \alpha\gamma}.$$

This equation can be simplified by collecting the constants.

Let $\epsilon = \beta\gamma$
$\rho = 1 + \alpha\gamma$

$$E = ct + c\gamma + e_0 + \frac{\beta t + \epsilon}{\rho + \alpha t}.$$

Dividing through by ϵ and letting

$$\lambda = \frac{\beta}{\epsilon} = \frac{1}{\gamma}$$
$$\mu = \frac{\rho}{\epsilon}$$
$$\nu = \frac{\alpha}{\epsilon}$$
$$E = ct + c\gamma + e_0 + \frac{\lambda t + 1}{\mu + \nu t}.$$

Dividing out the fraction,

$$E = ct + c\gamma + e_0 + \frac{\lambda}{\nu} + \frac{1}{\frac{\nu}{1 - \frac{\lambda\mu}{\nu}}t + \frac{\mu}{1 - \frac{\lambda\mu}{\nu}}}.$$

Let $M = c\gamma + e_0 + \frac{\lambda}{\nu}$

$$D = \frac{\nu}{1 - \frac{\lambda\mu}{\nu}}$$

$$B = \frac{\mu}{1 - \frac{\lambda\mu}{\nu}}.$$

Then $E = ct + M + \frac{1}{Dt + B}$

$$E = ct + M + \frac{1}{D(t + B/D)},$$

and letting $B/D = N$,

$$E = ct + M + \frac{1}{D(t + N)}. \qquad (5)$$

The equation now contains four constants which have the following values:

$$c = c$$
$$M = c\gamma + f_0 - e_0$$
$$D = -A$$
$$N = \frac{1}{Af_0} + \gamma.$$

It is obvious that some correlation exists between these constants; from equation (4)

$$e_0 = c\gamma + \frac{\beta\gamma}{1 + \alpha\gamma}$$
$$M = c\gamma + f_0 - c\gamma - \frac{\beta\gamma}{1 + \alpha\gamma} = \frac{f_0}{1 + Af_0\gamma}.$$
$$\frac{1}{M} = \frac{1}{f_0} + A\gamma$$
$$\frac{1}{MA} = \frac{1}{f_0 A} + \gamma = N$$
$$N = \frac{1}{MA} = -\frac{1}{DM}$$
or $$D = -\frac{1}{MN}.$$

Substituting this value in equation (5)

$$E = ct + M + \frac{MN}{t + N}$$
$$E = ct + M\frac{t}{t + N}. \qquad (6)$$

This equation is now in its simplest form. It contains three constants, c, M, and N; c is the slope of the elongation curve at infinite time; M is the total strain added during the test as illustrated in Fig. 11(C). N is a complex constant containing A and f_0 from the original Adams and Williamson equation and γ.

(2) Check of Equation for Elongation of a Glass Fiber

In deriving equation (6), the two assumptions were (1) that viscosity is constant and the change in rate of elongation is due to an elastic distortion which approaches a maximum value at a diminishing rate and (2) that this elastic distortion is equivalent to putting strain into the glass and is governed by the same laws as the rate of strain release during annealing. If these assumptions are correct, equation (6) should be the equation of the elongation-time curves. The three elongation curves selected to test this equation are illustrated in Fig. 12. The values of the constants in the equation were obtained for each curve by substituting values of E and t, taken from the curve, into three equations and solving them simultaneously for the constants. Three sets of such equations were solved for each curve, and the average of the three values of each constant thus obtained was taken as the true value of the constant. The circled points on the curves in Fig. 12 were calculated by using these constants in equation (6). Equation (6), therefore, is the true equation of the elongation-time curves for glass fibers under load. This check substantiates the

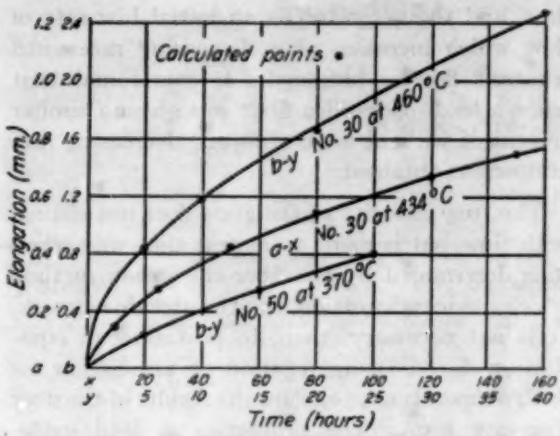

FIG 12.—Comparison of calculated and observed flow of glass fibers.

original assumption that the viscosity does not change with time and no equilibrium degree of aggregation need be postulated to explain the change of the rate of elongation with time.

(3) Effect of Thermal Treatment on Rate of Elongation

To establish the strain theory fully for the change of rate of elongation of glass fibers, the effect of thermal history on the shape of the elongation curve must be explained. Lillie pointed out that the normal shape of the elongation curve, when measured on a freshly drawn sample, is a relatively rapid flow at first, followed by a decreasing rate until a final constant value is reached. But if the fiber is held for several hours with the load in place at a temperature about 10°C below the test temperature and then suddenly heated to the test temperature, the initial rate of flow is small and it gradually increases to a constant value. He interpreted this reaction to indicate that if a glass is given a heat treatment at any other temperature than that of the test, it attains the equilibrium state of aggregation of that temperature, and time is required to change from this condition to the equilibrium state of the test temperature.

The elongation-time curves obtained on a fiber of glass No. 50 at 374°C are shown in Fig. 13. Curve No. 1 was obtained on the freshly drawn sample and is a normal elongation curve. Curve No. 2 was obtained on the same sample after it had been held at 364°C for 10 days with the load on the sample and then tested at 374°C. Curve No. 3 was obtained on the same fiber by removing the load and again lowering the temperature to 364°C. After soaking at this temperature for five days without the load, the temperature was rapidly raised to 374°C and the load was applied. Under these conditions the normal elongation curve was again obtained. This indicates that the change in the character of the elongation with heat treatment is dependent upon the load and not on a state of aggregation.

VII. Summary

On the basis of the atomic arrangement in lead oxide-silica glasses as derived from an X-ray investigation, which indicates that the glasses consist of a continuous, randomly-oriented network of silicon-oxygen tetrahedrons and lead atoms, an attempt was made to correlate the properties of these glasses and to give further enlightenment on the constitution of glass.

The "transformation point" was found to be unique when determined by some method which imposes no external force on the atomic network. It is defined as the temperature at which thermal vibration exceeds the strength of the most highly distorted atomic bonds which have previously attained the lowest energy content possible in the inherent distorted structure of glass. Above this temperature, a fraction of the atoms assumes an additional degree of freedom, that of transitional motion. Any external force which will continuously distort the atomic network or impose a directional force on some particular kind of atom in the structure will cause this temperature of spontaneous breakdown to be lowered or smeared beyond recognition. The transformation point is in accord with the continuous structure of glass.

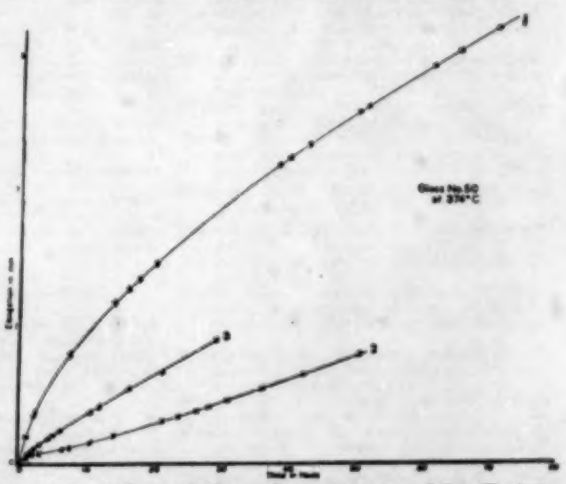

Fig. 13.—Effect of thermal treatment and loading on the elongation of glass fibers.

The theory of glass possessing an equilibrium degree of aggregation or association for each temperature is not consistent with the picture of a glass composed of a continuous, randomly oriented, atomic network. The aggregation theory is based chiefly on Lillie's study of the viscosity of glass by the fiber-elongation method. The experiments described in this paper show that the decreasing rate of elongation, with time, of a loaded, freshly drawn fiber is not due to a change in viscosity of the glass but to the decreasing rate at which "strain," or distortion of the atomic network, can be embodied in the glass structure. An equation of the rate of elongation of a glass fiber is derived by adapting the Adams and Williamson equation of the rate of strain release to the inverse condition, that of straining a glass. The check of this equation with experimental data obtained on the lead oxide-silica glasses substantiates this strain theory.

The strain theory likewise explains the fact that the fiber elongates at an increasing rate if, while loaded, it is heat-treated below the temperature of test for several hours and then rapidly heated to the test temperature. At the lower temperature, the fiber is capable of supporting greater continuous distortion than at the higher temperature and, if the equilibrium degree of distortion is attained at the lower temperature, it must assume the less distorted condition at the higher temperature and therefore must effectively contract. This contraction is superimposed on the constant viscous flow, and the net result is an initial low rate of flow which increases at a decreasing rate until constant flow is obtained. It was found that when a lead oxide-silica fiber was given a similar treatment without load, a normal decreasing rate of flow was obtained.

The true viscosity of the glass does not change with time but is made to appear that way when it is determined by the fiber-elongation method which induces distortion into the atomic network. It is not necessary, then, to postulate an equilibrium degree of aggregation or association for each temperature to explain the results of the fiber viscosity tests. The properties of lead oxide-silica glasses when they are tested by the fiber elongation method are, therefore, consistent with the picture of glass as accepted in this paper.

A glass is a continuous, randomly oriented, three-dimensional network of atoms.

Acknowledgments

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	Jan.	Feb.	March	April	May	June	July	Aug.	Sept.	Oct.	Nov.	Total
Abrasives	50	39	29	20	43	20	23	43	28	34	33	362
Art and Archeology	53	33	24	46	30	26	29	34	20	15	15	325
Cements	15	13	12	11	15	4	7	10	7	9	11	114
Enamel	71	28	48	19	30	14	32	36	30	19	62	389
Glass	130	91	71	88	79	91	87	186	76	78	92	1069
Structural Clay Products	36	17	16	11	9	12	13	13	15	14	8	164
Refractories	54	46	46	21	49	27	40	74	37	49	30	473
Terra Cotta	16	10	8	0	10	6	12	10	6	8	16	102
Whiteware	29	12	15	13	14	10	22	19	11	20	19	184
Equipment and Apparatus	55	39	45	39	48	40	38	45	43	39	37	468
Kilns, Furnaces, Fuels, and Combustion	33	17	25	15	24	15	33	41	20	27	30	280
Geology	78	16	22	37	49	26	30	22	17	18	36	351
Chemistry and Physics	57	44	38	33	34	34	42	60	35	49	25	451
General	41	12	20	21	12	24	14	18	14	28	17	221
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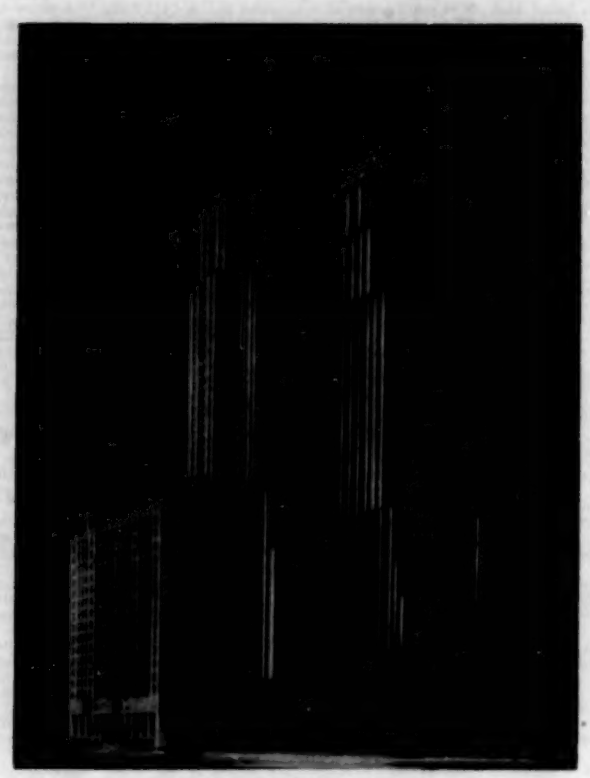

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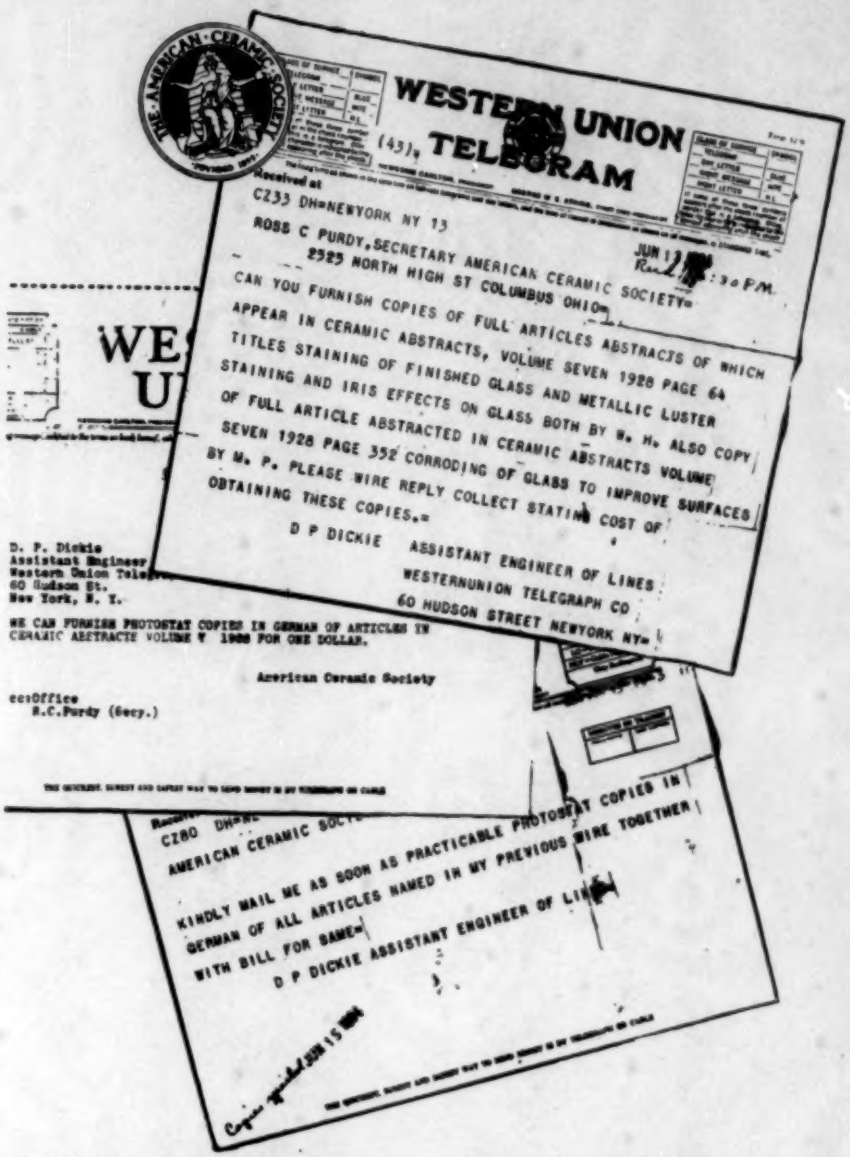
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